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IS 438 (2006): Aluminium powder for explosive and pyrotechnic compositions [CHD 26: Explosives and Pyrotechnics]



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भारतीय मानक
विस्फोटक और आतिशबाजी संघटन के लिए
एल्युमिनियम पाउडर — विशिष्टि
(दूसरा पुनरीक्षण)

Indian Standard

ALUMINIUM POWDER FOR EXPLOSIVES AND
PYROTECHNIC COMPOSITIONS — SPECIFICATION
(*Second Revision*)

ICS 71.060.01; 71.100.30

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

This standard was originally published in 1961. It was first revised in 1972 when the minimum limit of free metallic aluminium content for heavy grade of the material was raised from 96 to 98 percent. In the present revision, the grade is subdivided into Grade I and Grade II and metallic aluminium content are 98 and 90 percent respectively. For light grade, two sub-grades were included having different limits for free aluminium content and bulk density.

With a view to harmonize various standards formulated by different organizations like Defence, etc, for specific use, the Committee responsible for formulation of this standard decided to revise it to incorporate the requirements of other grades of material being used by Defence. Defence specification is more in line with light Grade 1. Hence in this revision, requirements for Defence purpose has been included. Requirement of water covering capacity is critical for its use in explosives and hence the same has been incorporated for light grade. Water covering capacity is the measure of surface area of flake. Aluminium powder when dispersed in water by having continuous layer of film on the surface of water. It gives an indicator of flat surface of the powder. Requirement for magnesium has also been included for light grade.

Aluminium powder, heavy, is used for the manufacture of aluminized explosives, incendiary compositions and pyrotechnic compositions. Aluminium powder, light, is used in explosives and pyrotechnic compositions. This standard is expected to assist the manufacturers and consumers in choosing the right quality of the material.

There is no ISO Standard on the subject.

The composition of the Committee responsible for the formulation of this standard is given in Annex P.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ALUMINIUM POWDER FOR EXPLOSIVES AND PYROTECHNIC COMPOSITIONS — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for aluminium powder for use in explosive and pyrotechnic compositions.

2 REFERENCES

The following standards listed below contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
170 : 1986	Specification for acetone (<i>third revision</i>)
264 : 1976	Nitric acid (<i>second revision</i>)
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
266 : 1993	Sulphuric acid (<i>third revision</i>)
323 : 1959	Specification for rectified spirit (<i>revised</i>)
878 : 1975	Graduated measuring cylinders (<i>first revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
4905 : 1968	Methods for random sampling

3 GRADES

3.1 There shall be two grades of the material as follows:

- a) Aluminium powder, light; and
- b) Aluminium powder, heavy (also known as 'blown' aluminium powder).

3.1.1 Both the grades of aluminium powder shall be of two sub-grades, namely, sub-grade I and sub-grade II depending upon its free metallic aluminium content and bulk density.

4 REQUIREMENTS

4.1 Description

4.1.1 *Aluminium Powder, Light* — The material shall be in the form of flakes, free from visible impurities.

4.1.2 *Aluminium Powder, Heavy* — The material shall be in the form of compact, irregular and elongated particles (not flakes). It shall be free from visible impurities and foreign matter. An aqueous extract of the material shall not be alkaline to phenolphthalein.

4.2 **Particle Size** — The material shall conform to the sieving requirements as agreed to between the purchaser and the supplier.

4.3 Gritty Matter

4.3.1 *Aluminium Powder, Light* — The material of sub-grade I shall contain not more than 0.01 percent by mass of gritty matter when determined as prescribed in Annex A. The gritty matter shall completely pass through 125-micron IS Sieve. In case of sub-grade II, it shall not be traceable.

4.3.2 *Aluminium Powder, Heavy* — The material of sub-grade I shall contain not more than 0.05 percent by mass (for sub-grade II not more than 0.1 percent by mass) if gritty matter when determined as prescribed in Annex A. The gritty matter shall completely pass through 425-micron IS Sieve.

4.4 The material shall also comply with the requirement given in Table 1 when tested according to the methods prescribed in Annexes B to M. Reference to the relevant annexes is given in col 7 of Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in steel drums painted, both inside as well as outside, with aluminium paint or in galvanized steel drums.

5.1.1 When the material is required to be transported by rail, the packing shall conform to the provisions of Indian Railways Conference Association, Red Tariff No. 18.

CAUTION: STORE THE DRUMS OF FLAKES/
POWDER IN DRY PLACE

5.2 Marking

The drums shall be legibly and indelibly marked with the following information.

- Name, grade and sieve analysis of the material;
- Manufacturer's name and/or his trade-mark, if any;
- Identification, in code or otherwise, to enable the lot of manufacture to be traced back from the records;
- Tare and net mass; and
- Year and month of manufacture.

5.2.1 BIS Certification Marking

The packages may also be marked with the BIS Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The

details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex N.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirement for Aluminium Powder for Explosives and Pyrotechnic Compositions

(Clause 4.4)

Sl No.	Characteristic	Requirement				Methods of Test, Ref to Annex
		Light Grade		Heavy Grade		
		I	II	I	II	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Matter soluble in ether, percent by mass, <i>Max</i>	2.0 ¹⁾	2.0 ¹⁾	0.25 ¹⁾	0.25 ¹⁾	B
ii)	Volatile matter, percent by mass, <i>Max</i>	0.25-0.50	0.25-0.50	0.05	0.05	C
iii)	Silicon (as Si), percent by mass, <i>Max</i>	0.5	0.5	0.5	0.5	D
iv)	Copper (as Cu), percent by mass, <i>Max</i>	0.1	0.1	0.25	0.25	E
v)	Iron (as Fe), percent by mass, <i>Max</i>	1.0 ¹⁾	1.0 ¹⁾	1.0 ¹⁾	1.0 ¹⁾	F
vi)	Zinc (as Zn), percent by mass, <i>Max</i>	0.1	0.1	0.25	0.25	G
vii)	Magnesium (as Mg), percent by mass, <i>Max</i>	0.2	0.2	0.2	0.2	H
viii)	Other metallic impurities (as Na), percent by mass, <i>Max</i>	1.0	1.0	0.5	0.5	J
ix)	Free metallic aluminium, percent by mass, <i>Min</i>	87.0	85.0	98.0	90.0	K
x)	Apparent density, g/ml, <i>Max</i>	0.3	0.5	a) for material passing through 2.00 mm IS Sieve but retained on 425-micron IS Sieve: 0.9, <i>Min</i> b) for material of other sizes: 1.0, <i>Min</i>		L
xi)	Water covering, capacity cm ² /g, <i>Min</i> (for use in commercial explosives)	9 000 ²⁾	—	—	—	M

¹⁾ For special requirements, the limits may be as agreed to between the purchaser and the supplier.

²⁾ Not applicable for fireworks.

ANNEX A

(Clauses 4.3.1 and 4.3.2)

DETERMINATION OF GRITTY MATTER

A-1 REAGENTS

A-1.1 Concentrated Hydrochloric Acid — See IS 265.

A-1.2 Sodium Hydroxide Solution — Dissolve 50 g of sodium hydroxide in 100 ml of water.

A-1.3 Dilute Hydrochloric Acid — 2 percent (v/v).

A-2 PROCEDURE

A-2.1 Dissolve about 25.0 g of the material, accurately weighed, in sufficient concentrated hydrochloric acid and boil until the metallic particles have dissolved and the solution becomes clear. Filter through a filter paper Whatman No. 40 or its equivalent. Wash the filter paper thoroughly, dry it and ignite gently. Boil the ignited residue for exactly 10 min with 20 ml of sodium hydroxide solution. Dilute immediately with 200 to 300 ml of water. Wash the gritty matter thrice by decantation and then once with dilute hydrochloric

acid. Filter the gritty matter through a filter paper (Whatman No. 40 or its equivalent), wash it and ignite gently. Transfer the residue to 63-micron IS Sieve and weigh the material retained on the sieve as gritty matter.

A-2.1.1 Sieve the weighed residue through the relevant IS Sieve specified in 4.3.1 or 4.3.2 using a small soft brush.

A-3 CALCULATION

$$\text{Gritty matter, percent by mass} = \frac{100 M_1}{M}$$

where

M_1 = mass of the gritty matter as obtained in A-2.1, in g; and

M = mass of the material taken for the test, in g.

A-3.1 In the report, state if any residue is left on the relevant sieve specified in 4.3.1 or 4.3.2.

ANNEX B

[Clause 4.4 and Table 1, Sl No. (i)]

DETERMINATION OF MATTER SOLUBLE IN ETHER

B-1 REAGENTS

B-1.1 Dilute Hydrochloric Acid — 1 : 1 (v/v).

B-1.2 Diethyl Ether

B-1.3 Methyl Orange Indicator — 0.1 percent in water.

B-1.4 Acetone — See IS 170.

B-2 PROCEDURE

B-2.1 Weigh accurately about 2 g of the material into a 400-ml beaker, add 100 ml of hot water and then add 60 ml of dilute hydrochloric acid in small portions, allowing the reaction to reach completion after each addition. Cover the beaker with a watch glass and boil the contents of the beaker on a hot plate until all the aluminium has dissolved. Cool the contents of beaker. Wash the contents first with diethyl ether and then with water into a separating funnel. Further, wash the beaker with water and ether and transfer all the washings to the separating funnel.

B-2.2 Shake the contents of the separating funnel for 1 min and allow to stand until the ether and water layers have separated. Then run off the water layer and collect

the ether layer. Extract the aqueous layer twice with ether in the same way and collect ether layer. Wash the combined ether layer in the separating funnel with further quantities of water until the washings are free from acid (as indicated by methyl orange indicator).

B-2.3 Run off carefully as much of the water as possible from the separating funnel and transfer the ether layer to a tared 100-ml beaker. Wash the funnel with acetone to ensure complete transfer of oily and fatty matter. Gently warm the beaker and its contents on a water-bath until the ether has been evaporated as completely as possible. Continue the evaporation by heating the beaker in an oven at a temperature of $105 \pm 2^\circ\text{C}$ for 1 h, cool and weigh till a constant mass is obtained.

B-3 CALCULATION

$$\text{Matter soluble in ether, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass of the residue obtained, in g; and

M = mass of the sample taken for the test, in g.

ANNEX C

[Clause 4.4 and Table 1, Sl No. (ii)]

DETERMINATION OF VOLATILE MATTER

C-1 PROCEDURE

Heat a flat-bottom glass dish, of about 5 cm diameter provided with a ground-glass cover, in an oven maintained at 100 to 105°C for 30 min. Cool in a desiccator and weigh. Place about 5 g of the material in the dish. Replace the cover and weigh accurately. Uncover the dish and heat for 1 h in the oven. Remove the dish, replace the cover, cool in a desiccator and weigh accurately.

C-2 CALCULATION

$$\text{Volatile matter, percent by mass} = \frac{100 (M_1 - M_2)}{M}$$

where

M_1 = mass of the dish with the material before heating, in g;

M_2 = mass of the dish with the material after heating, in g; and

M = mass of the material taken for the test, in g.

ANNEX D

[Clause 4.4 and Table 1, Sl No. (iii)]

DETERMINATION OF SILICON

D-1 REAGENTS

D-1.1 Concentrated Hydrochloric Acid—See IS 265.

D-1.2 Dilute Hydrochloric Acid — 2 percent (v/v).

D-1.3 Dilute Sulphuric Acid — 40 percent and 2 percent (v/v).

D-1.4 Ammonium Acetate Solution — 20 percent (m/v) approximately.

D-1.5 Aqua Regia — Mix three volume of concentrated hydrochloric acid with one volume of concentrated nitric acid.

D-1.6 Sodium Hydroxide Solution — 50 percent and 5 percent approximately.

D-2 PROCEDURE

D-2.1 Place 2 g of the material, accurately weighed, in a 600-ml beaker and add 50 ml of water. Then add 5 ml of concentrated hydrochloric acid. Warm the liquid until the reaction starts. When the first violent evolution of gas has subsided, add 20 ml more of concentrated hydrochloric acid in small portions. When almost all the solid has dissolved, add 30 ml of dilute sulphuric acid (40 percent) and evaporate the liquid on a sand tray or hot-plate until copious fumes are evolved. Cool the residue, dilute with 200 ml of water and warm with occasional stirring. Allow the contents of the beaker to stand overnight, filter off the insoluble residue next morning through a filter paper (Whatman No. 42 or its equivalent) and wash with dilute sulphuric acid (2 percent) several times. Reserve the filtrate for determination of iron (see Annex F).

D-2.2 Extract the insoluble residue in a 100-ml beaker with 20 ml of hot ammonium acetate solution. After 30 min of extraction, filter the liquid through a filter paper (Whatman No. 41 or its equivalent) into a 100 ml beaker and wash the residue once with ammonium acetate solution. Dry the residue, ignite in a porcelain crucible and weigh (M_1). Warm the ignited residue with 10 ml of aqua regia for 5 min, dilute the solution, and filter it through a filter paper (Whatman No.40 or its equivalent). Wash the residue with water, dry it and ignite gently. Transfer the ash to a 100-ml beaker made of resistant glass, add 20 ml of sodium hydroxide solution (50 percent) and boil gently for 10 min. Wash the residue and the solution into a tall 400-ml beaker containing 200-ml of water. Allow the solid matter to settle for 15 min, siphon or decant off the clear liquid and add a further 200 ml of water. Repeat the washing by decantation once more and then filter off the residue on a filter paper (Whatman No.40 or its equivalent). Wash the residue on the paper with hot water and finally with dilute hydrochloric acid. Dry the residue, ignite in a porcelain crucible and weigh the ash (M_2). The loss in mass ($M_1 - M_2$) is due to silica derived from silicon originally present as such.

D-3 CALCULATION

$$\text{Silicon (as Si), percent by mass} = \frac{46.70 (M_1 - M_2)}{M}$$

where

M_1 = mass of the residue from ammonium acetate extraction, in g;

M_2 = mass of the ignited residue, in g; and

M = mass of the material taken for the test, in g.

ANNEX E

[Clause 4.4 and Table 1, Sl No. (iv)]

DETERMINATION OF COPPER

E-1 APPARATUS

E-1.1 Nessler Cylinder — 50 ml capacity.

E-2 REAGENTS

E-2.1 Sodium Hydroxide Solution — 10 percent approximately.

E-2.2 Dilute Nitric Acid — 70 percent approximately.

E-2.3 Ammonium Nitrate Solution — 500 g/l.

E-2.4 Phenolphthalein Indicator Solution — 0.1 g in 100 ml of 60 percent rectified spirit.

E-2.5 Ammonium Hydroxide — 20 percent approximately.

E-2.6 Sodium Diethyldithiocarbamate Solution — Dissolve 0.1 g of the reagent in water. Filter, if necessary. Preserve in an amber coloured glass bottle and store in a dark place.

E-2.7 Carbon Tetrachloride

E-2.8 Dilute Sulphuric Acid — 10 percent approximately.

E-2.9 Standard Copper Solution — Dissolve 3.928 g of copper sulphate, penta hydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and dilute the solution to exactly one litre. Dilute 10 ml of this solution to one litre with water. One millilitre of this solution contains 0.01 mg of copper (as Cu). The diluted solution should be prepared immediately before use.

E-3 PROCEDURE

E-3.1 Weigh accurately 2.0 g of the material in a 600 ml beaker. Add 100 ml sodium hydroxide solution, cover with a watch glass and allow to stand till the reaction subsides. Boil gently for a few minutes to

complete the reaction, cool and add 50 ml of dilute nitric acid and boil to complete solution. Cool, filter if necessary, make up to 250 ml in a volumetric flask with water and mix the solution well.

E-3.2 Transfer by pipette 10 ml of the sample solution to a 400-ml beaker, add 10 ml of ammonium nitrate solution, 0.2 ml of phenolphthalein indicator and, slowly with intermittent shaking, ammonium hydroxide until the solution just becomes pink. Transfer the solution to a small separating funnel and add 20 ml of sodium diethyldithiocarbamate solution. Extract the solution with successive 10 ml portions of carbon tetrachloride until the solvent layer remains colourless, transferring each extract into 50-ml Nessler cylinder. Dilute the extract to 50-ml with carbon tetrachloride. Compare the colour with that of standard solutions of copper prepared as in E-3.3.

E-3.3 Take 20 ml of the standard copper solution in a 400 ml beaker and dilute to 25 ml with water. Add one drop of dilute sulphuric acid and 3 drops of phenolphthalein indicator, followed by slow addition of ammonium hydroxide until the solution just becomes pink. Treat the solution with sodium diethyldithiocarbamate solution and extract the resultant copper salt with carbon tetrachloride in the same manner as in the case of sample solution. Transfer the extracts to Nessler cylinder and dilute to 50 ml mark with carbon tetrachloride.

E-3.4 In case of light grade of the material, use 5.0 g of the material for the test.

E-3.5 The limits prescribed in Table 1 shall be considered to have not exceeded, if the intensity of the colour produced by the material is not greater than that produced in the control test.

ANNEX F

[Clause 4.4 and Table 1, Sl No. (v)]

DETERMINATION OF IRON

F-1 REAGENTS

F-1.1 Concentrated Nitric Acid — See IS 264.

F-1.2 Sodium Hydroxide Solution — 30 percent approximately.

F-1.3 Dilute Hydrochloric Acid — 1 : 1 (v/v).

F-1.4 Stannous Chloride Solution — Dissolve 6 g of stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in 6 ml of concentrated hydrochloric acid and dilute the solution to 100 ml.

F-1.5 Mercuric Chloride Solution — saturated.

F-1.6 Diphenylamine Indicator — Dissolve 0.1 g of

diphenylamine in 100 ml of concentrated sulphuric acid.

F-1.7 Standard Potassium Dichromate Solution — 0.1 N.

F-2 PROCEDURE

Pass hydrogen sulphide gas through the filtrate reserved in D-2.1 for 20 min, filter off the precipitate on a filter paper (Whatman No. 41 or its equivalent). Boil the filtrate to remove hydrogen sulphide. Then add 3 ml of concentrated nitric acid and boil for about 2 min. Allow the liquid to cool a little and then dilute the solution to about 300 ml; while the solution is still hot, add sodium hydroxide solution with vigorous stirring until all the aluminium hydroxide, first precipitated, is just redissolved. (The amount of sodium hydroxide solution necessary is about 60 ml.) Filter off the brown precipitate of ferric hydroxide on a filter paper (Whatman No. 54 or its equivalent). Reserve the filtrate for determination of zinc as described in Annex G. Wash the precipitate, once with hot water and dissolve it in about 30 ml of warm dilute hydrochloric acid. Heat the solution to the boiling point and add

stannous chloride solution drop by drop until the yellow colour, due to ferric iron just disappears. After cooling, add 5 ml of mercuric chloride solution in order to oxidize the slight excess of stannous chloride. Allow the liquid to stand for a few minutes, add two or three drops of diphenylamine indicator and titrate with potassium dichromate solution. The end point is indicated by the appearance of deep blue colour. Carry out a blank titration using same quantities of all the reagents used and apply the necessary correction to the original titration.

F-3 CALCULATION

$$\text{Iron (as Fe), percent by mass} = 5.6 \frac{V N}{M}$$

where

V = volume of standard potassium dichromate solution used in the titration, in ml;

N = normality of standard potassium dichromate solution; and

M = mass of the material taken for test in D-2.1, in g.

ANNEX G

[Clause 4.4 and Table 1, Sl No. (vi)]

DETERMINATION OF ZINC

G-1 REAGENTS

G-1.1 Hydrogen Sulphide — gas.

G-1.2 Hydrogen Sulphide Solution — saturated.

G-1.3 Dilute Hydrochloric Acid — 1 : 1 (v/v).

G-1.4 Dilute Ammonium Hydroxide — 1 : 4 (v/v).

G-1.5 Dilute Acetic Acid — 1 : 4 (v/v).

G-1.6 Ammonium Nitrate Solution — saturated.

G-2 PROCEDURE

Pass hydrogen sulphide slowly for 15 min into the filtrate reserved in F-2 and set the liquid aside for 2 h in order to allow the precipitate, if any, to settle. Filter off the precipitate on a filter paper (Whatman No. 54 or its equivalent) and wash once with hydrogen sulphide solution. Dissolve the precipitate on the filter paper, in 20 ml of warm dilute hydrochloric acid and wash with water into a 150-ml beaker. Boil the solution gently to remove hydrogen sulphide. Add dilute ammonium hydroxide to the solution until it is just

alkaline to litmus paper, boil for a minute or two and filter off any precipitate of aluminium hydroxide on a small filter paper (Whatman No. 41 or its equivalent). After washing twice with hot water, discard the residue and make the filtrate just acidic with dilute acetic acid. Saturate the solution with hydrogen sulphide, allow the precipitate to settle, and filter it on a filter paper (Whatman No. 40 or its equivalent). Wash the precipitate once with hydrogen sulphide solution, moisten the paper with a few drops of ammonium nitrate solution, dry, ignite, cool in a desiccator and weigh till constant mass is obtained.

G-3 CALCULATION

$$\text{Zinc (as Zn), percent by mass} = 80.34 \frac{M_1}{M}$$

where

M_1 = mass of the ignited residue, in g; and

M = mass of the material taken for test in D-2.1, in g.

ANNEX H

[Clause 4.4 and Table 1, *Sl No. (vii)*]

DETERMINATION OF MAGNESIUM

H-1 REAGENTS

H-1.1 Sodium Hydroxide Solution — 20 percent (*m/v*), 0.2 percent (*m/v*) and 2 N.

H-1.2 Aqua Regia — Mix three volumes of concentrated hydrochloric acid with one volume of concentrated nitric acid.

H-1.3 Sodium Tartrate Solution — 20 percent (*m/v*).

H-1.4 Phenolphthalein Indicator Solution — Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent of rectified spirit.

H-1.5 Potassium Cyanide Solution — 30 percent (*m/v*).

H-1.6 8-Hydroxyquinoline (Oxine) Solution — Dissolve 5 g of oxine in 12 ml of glacial acetic acid and dilute to 100 ml with water or dissolve 2 g of oxine in 100 ml of 2 N acetic acid.

H-1.7 Rectified Spirit — 95 percent by volume (*see* IS 323).

H-1.8 Dilute Hydrochloric Acid — 1 : 1 (*v/v*).

H-1.9 Indigo Carmine Solution — 1 percent.

H-1.10 Standard Potassium Bromate-Potassium Bromide Solution — 0.1 N.

H-1.11 Potassium Iodide Solution — 10 percent (*m/v*).

H-1.12 Starch Solution — Tritrate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into 1 litre of boiling water. Boil for 3 min. Allow the solution to cool and decant off the supernatant clear liquid.

H-1.13 Standard Sodium Thiosulphate Solution — 0.1 N.

H-2 PROCEDURE

H-2.1 Heat 2 g of the material, accurately weighed, with 40 ml of sodium hydroxide solution (20 percent), dilute to 150 ml and boil. Filter off the residue on a prepared Gooch crucible and wash with hot sodium hydroxide solution (0.2 percent). Extract the residue with 15 ml of hot diluted (1:1) aqua regia and wash with hot water. Combine the extract and the washings and make up with water to 100 ml in a volumetric flask.

H-2.2 Pipette 10 ml of this solution into a 150 ml

beaker, add 5 ml of water, 5 ml of sodium tartrate solution and one drop of phenolphthalein indicator. Neutralize with sodium hydroxide solution (2 N) and add 2 ml in excess. Add 2 ml of potassium cyanide solution and 2 ml of oxine solution. Stir to induce precipitation and then stir for a further period of 1 min. Allow to stand for 10 min and again stir for 1 min. Heat to 60°C and cool.

H-2.3 Filter off the precipitate with the aid of suction on a sintered glass crucible (G No. 3 or its equivalent) and wash in ten portions with 120 ml of solution prepared by mixing 500 ml of rectified spirit, 430 ml of water, 50 ml of sodium tartrate solution and 20 ml of sodium hydroxide solution (2 N). Extract the precipitate with 20 ml of hot dilute hydrochloric acid and 30 ml of hot water, collecting the extract on a conical flask. Cool, add 3 to 4 drops of indigo carmine solution, add slowly from a burette, bromate-bromide solution in order to provide an excess. Note the total quantity of bromate-bromide solution added (V_1), and without delay add 10 ml of potassium iodide solution and a few drops of freshly prepared starch solution and titrate the liberated iodine with standard sodium thiosulphate solution until the blue colour is just discharged. Note the volume of thiosulphate solution used.

H-2.4 Carry out a control determination, as prescribed in H-2.2 and H-2.3 of another 10 ml portion of the solution prepared in H-2.1 to which has been added 2 ml of a solution containing 2.05 g of magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) per litre. This will be equivalent to the addition of 0.2 percent magnesium to the sample. The difference between the two percentages of magnesium thus obtained shall be exactly equal to the added percentage of magnesium.

H-3 CALCULATION

$$\text{Magnesium (as Mg), percent by mass} = \frac{0.304 (V_1 - V_2)}{M}$$

where

V_1 = total volume of standard bromate-bromide solution added, in ml;

V_2 = volume of standard sodium thiosulphate solution used for titration, in ml; and

M = mass of the material taken for the test, in g.

ANNEX J

[Clause 4.4 and Table 1, *Sl No.* (viii)]

DETERMINATION OF OTHER METALLIC IMPURITIES

J-1 REAGENTS

J-1.1 Concentrated Hydrochloric Acid — See IS 265.

J-1.2 Concentrated Nitric Acid — See IS 264.

J-1.3 Ammonium Chloride

J-1.4 Dilute Ammonium Hydroxide — 1 : 4 (v/v).

J-1.5 Concentrated Sulphuric Acid — See IS 266.

J-2 PROCEDURE

J-2.1 Place about 0.5 g of the material, accurately weighed, in 600-ml beaker, cover with 50 ml of warm water and add concentrated hydrochloric acid gradually until the material has dissolved (10 ml of acid is usually sufficient). Add 2 ml of concentrated nitric acid to oxidize iron, evaporate the solution to dryness and remove nitric acid by a further evaporation after addition of 5 ml of concentrated hydrochloric acid. Heat the residue obtained at about 120°C for an hour, cool and dissolve in 10 ml of concentrated hydrochloric acid and dilute with 300 ml of water. Add 5 g of ammonium chloride, boil the solution and add dilute ammonium hydroxide in slight excess. Boil for a further 5 min after which filter off the precipitates of aluminium and iron hydroxides on a filter paper (Whatman No. 41 or its equivalent). Wash the precipitate with hot water containing 2 percent of ammonium chloride. Evaporate the filtrate and washings to about 50 ml in a beaker made of resistant

glass, make just acidic with concentrated hydrochloric acid, boil for few minutes and then make it just alkaline with dilute ammonium hydroxide in order to precipitate any aluminium left in the solution. Filter through a filter paper (Whatman No. 41 or its equivalent), evaporate the filtrate to dryness in a beaker made of resistant glass, transfer the solid residue to a tared platinum crucible, using moist filter paper to transfer the last traces of solid. Burn the paper and volatilize the solid ammonium chloride by gentle heating. Sulphate the residue with two drops of concentrated sulphuric acid, ignite at red heat, cool and weigh.

J-2.2 Carry out a blank determination using all the reagents in the same manner. Then correct the mass of the sulphated residue for the amount of residue found in the blank determination and for the amounts of copper and zinc as determined in Annex E and Annex G respectively, assuming these to be present as oxides (CuO and ZnO). Regard the remainder (M_1) as sodium sulphate and express the result in terms of sodium (Na).

J-3 CALCULATION

Other metallic impurities

$$(\text{as Na}), \text{ percent by mass} = 32.38 \frac{M_1}{M_2}$$

where

M_1 = mass of the residue as obtained in J-2.2, in g; and

M_2 = mass of the material taken for test, in g.

ANNEX K

[Clause 4.4 and Table 1, *Sl No.* (ix)]

DETERMINATION OF FREE METALLIC ALUMINIUM

K-1 APPARATUS

The apparatus shall consist of the following main parts assembled as shown in Fig. 1.

K-1.1 Lunge Nitrometer — provide with water jacket.

K-1.2 Reaction Bottle — capacity about 60 ml.

K-1.3 Weighing Tube — closed at one end, about 4 cm long and 0.5 cm internal diameter.

K-2 REAGENTS

K-2.1 Sodium Hydroxide Solution — 10 percent (m/v).

K-3 PROCEDURE

Weigh accurately about 0.1 g of the material into the weighing tube. Introduce the tube containing the material into the reaction bottle. Add carefully through a funnel 20 ml of sodium hydroxide solution to the reaction bottle and connect it to the Lunge

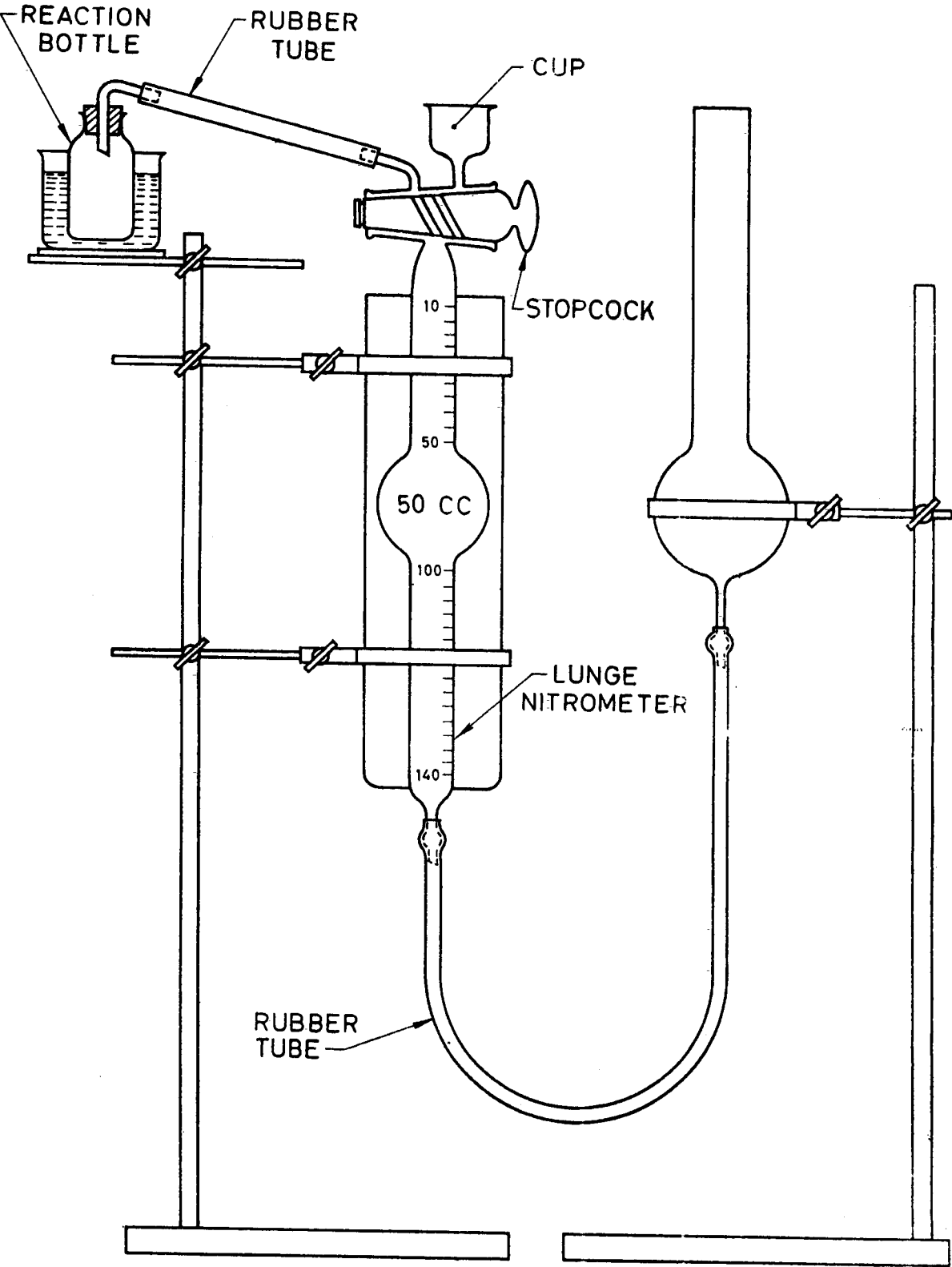


FIG. 1 APPARATUS FOR DETERMINATION OF FREE METALLIC ALUMINIUM

nitrometer filled with acidulated water, with the due regard for air-tightness. Make sure that the air in the reaction bottle is initially at atmospheric pressure. Expel all the air completely from the nitrometer bulb by bringing up the level of the acidulated water coloured with a drop of methyl red. Note the temperature of the water jacket around the nitrometer and of the water-bath around the reaction bottle, which should almost be the same. Tilt the reaction bottle in such a way that the sodium hydroxide solution finds its way into the weighing tube containing the material and at the same time open the passage to the nitrometer. When the reaction ceases, adjust the pressure very nearly to the atmospheric pressure and allow the apparatus to stand for 1 h. Add a drop or two of methyl red in the cup of the nitrometer to serve as an index for the pressure of the gas in the nitrometer. Find out by trial, the position at which the liquid in the nitrometer cup does not move up or down the bore of the stopper when the nitrometer is open to atmosphere. This operation is to be repeated after putting the gas in the nitrometer in communication with that in the reaction bottle, to ensure that the air closed in the latter is at atmospheric pressure. Read the volume of the gas. Read the temperature of the water jacket and the water-bath

around the reaction bottle. Note the barometric height and the temperature.

NOTES

1 Correct the volume of hydrogen for the volume of gas produced by metallic zinc only. One hundred grams of pure metallic zinc will produce 34 253 ml of hydrogen, measured at NTP. (This correction may be ignored if metallic zinc present in the sample is less than 0.1 percent.)

2 One hundred grams of pure metallic aluminium will produce 124 580 ml of hydrogen, measured at NTP.

K-4 CALCULATION

$$\text{Free metallic aluminium, percent by mass} = \frac{V(P-p) \times 273 \times 10^4}{(273+t) \times 760 \times 124\,580 \times M}$$

where

V = volume of hydrogen gas produced, in ml;

P = atmospheric pressure of mercury at the instant of measurement of volume of hydrogen gas produced, in mm;

p = vapour pressure of mercury of acidulated water at $t^\circ\text{C}$, in mm;

t = temperature at the instant of measurement of the volume of hydrogen gas produced, in $^\circ\text{C}$; and

M = mass of the material taken for the test, in g.

ANNEX L

[Clause 4.4 and Table 1, Sl No. (x)]

DETERMINATION OF APPARENT DENSITY

L-1 APPARATUS

L-1.1 Measuring Cylinder — 100 ml capacity, stoppered, conforming to IS 878.

L-1.2 Stand — Suitable stand arrangement for the cylinder to fall vertically through a distance of 6.5 cm on to a hard leather pad.

L-2 REAGENT

L-2.1 Rectified Spirit — 95 percent (v/v) (see IS 323).

L-3 PROCEDURE

L-3.1 Aluminium Powder, Light

Add about 15 g of the material to 60 ml of rectified spirit contained in the stoppered measuring cylinder. Then gently shake for about 5 min to ensure thorough mixing. Wash down the material on the sides of the measuring cylinder and the stopper into the bulk by means of a jet of rectified spirit from a wash bottle and then allow the cylinder to stand for a minimum

of 2 h, after which read the volume occupied by the material.

L-3.2 Aluminium Powder, Heavy

Introduce 20 g of the material into the stoppered measuring cylinder. Drop the cylinder vertically 30 times from a height of 6.5 cm on to a hard leather pad. Level the material by the minimum amount of tapping on the sides of the cylinder and read the volume occupied by the material.

L-4 CALCULATION

$$\text{Apparent density, g/ml} = \frac{M}{V}$$

where

M = mass of the material taken for the test, in g; and

V = volume occupied by the material, in ml.

ANNEX M

[Clause 4.4 and Table 1, Sl No. (xi)]

DETERMINATION OF WATER COVERING CAPACITY

M-1 APPARATUS

M-1.1 Water Tray (Coverage Determination Tray)

— Rectangular in shape and approximately 650 mm long, internal width 120 mm and 13 to 15 mm deep with vertical sides about 13 mm thick, machined and finished smooth on the upper surface.

M-1.2 Plastic Wash Bottles (Squeeze Bottles)

M-2 REAGENTS

M-2.1 2-Butanol

M-2.2 Paraffin Wax

M-3 PROCEDURE

Weigh, accurately 15 to 20 mg of aluminium powder, to the nearest of fourth place of decimal, in a watch glass. Care should be taken such that no air current shall come across the sample material while weighing and handling.

Wax is rubbed on the sides of the coverage determination tray (placed on a level surface checked with a spirit level) and glass plates, to avoid overflow of water.

Add two drops of 2-butanol to it and make a paste with the help of a glass rod. Add another one to two drops of 2-butanol and transfer the whole mass of paste, quantitatively, between the two glass plates of the coverage tray filled with water, with the help of a jet of water from wash bottle, with extreme care. Slowly, shake the film formed on the water level, with the glass rod, so that all the agglomerates of aluminium break into a homogeneous film. Adjust the glass plates till wrinkles just occur in the film. Finally, measure the distance between the two plates, at which the wrinkles formed on the surface just disappear without any break or gap in the film.

M-4 CALCULATION

Water covering capacity of
the powder (in cm²/g) = $\frac{L_1 \times L_2}{W}$

where

L_1 = inner width, of the tray/trough, in cm;

L_2 = final distance, between the two plates, in cm;
and

W = weight of the sample, in g.

ANNEX N

(Clause 6)

SAMPLING OF ALUMINIUM POWDER FOR EXPLOSIVE AND
PYROTECHNIC COMPOSITIONS

N-1 GENERAL REQUIREMENTS OF SAMPLING

N-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

N-1.2 Samples shall not be drawn in an exposed place.

N-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contaminations.

N-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

N-1.5 The samples shall be placed in clean, dry, air-tight glass or other suitable containers which have no action on the material.

N-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

N-1.7 Each sample container shall be sealed air-tight with a stopper after filling, and marked with all particulars of the material (see 5.2) and the date of sampling.

N-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the ambient temperature.

N-2 SCALE OF SAMPLING

N-2.1 Lot

In any consignment, all the containers of the same grade and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to

consist of different grades or batches of manufacture, the containers belonging to the same grade and batch shall be grouped together and each such group shall constitute a separate lot.

N-2.1.1 For ascertaining the conformity of the material in a lot to the requirements of the specification, test shall be carried out for each lot separately. The number of containers to be selected from a lot shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling

Lot Size <i>N</i> (1)	Sample Size <i>n</i> (2)
3 to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

N-2.2 The containers shall be selected at random, from a lot. In order to ensure randomness of selection, some random number table (*see* IS 4905) as agreed to between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure is recommended for use:

Arrange the containers in the lot in a systematic manner and starting from any one, count them as 1, 2..., etc, up to *r* and so on where *r* is the integral part of *N/n* (*N* being the lot size and *n* the sample size, respectively). Every *r* th container thus counted shall be withdrawn to constitute the sample.

N-3 PREPARATION OF TEST SAMPLE

N-3.1 From each of the containers selected according to N-2.2, a small representative portion of the material not less than 200 g shall be taken out.

N-3.2 Out of these portions, small but equal quantities of the material shall be taken and thoroughly mixed to form a composite sample, not less than 150 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

N-3.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of the individual samples representing the *n* containers shall be marked for the purchaser, another for the supplier and third to be used as referee sample.

N-3.4 All the individual samples and composite samples shall be transferred to separate sample containers. The sample containers shall be sealed and labelled with full identification particulars (*see* N-1.7).

N-3.5 The referee samples consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier, to be used in the case of any dispute between the two parties.

N-4 NUMBER OF TESTS

N-4.1 Tests for the determination of gritty matter and the volatile matter shall be performed on the individual sample.

N-4.2 Tests for all other characteristics given in 4 shall be performed on the composite sample.

N-5 CRITERIA FOR CONFORMITY

N-5.1 For Individual Samples

From each set of test results for gritty matter and volatile matter, the mean (\bar{X}) and range (*R*) of test results shall be computed separately (the range being defined as the difference between the maximum and the minimum values of the test results).

N-5.1.1 The lot shall be declared as conforming to the requirements of gritty matter and volatile matter if, $\bar{X} + 0.6 R$ as calculated from the relevant test results is less than or equal to the relevant maximum value specified in 4.

N-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement specified in Table 1.

ANNEX P

(Foreword)

COMMITTEE COMPOSITION

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